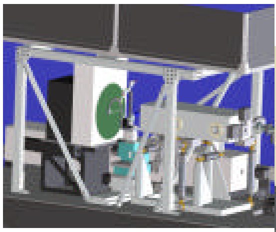
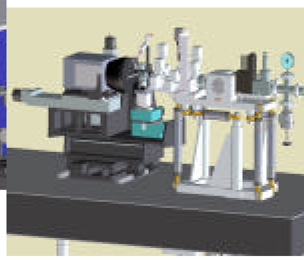
West Coast High-Pressure Facilities
Current Status

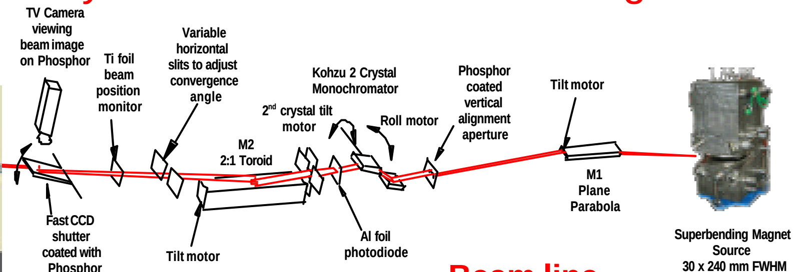
Beamline 12.2.2 High-pressure x-ray diffraction with laser heating

End station 1

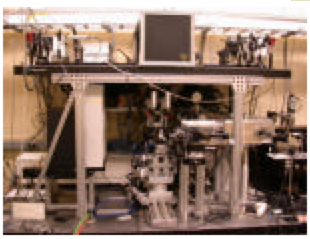


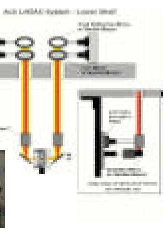


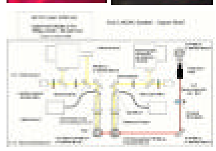
End station 2



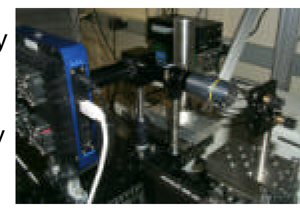
Beam line







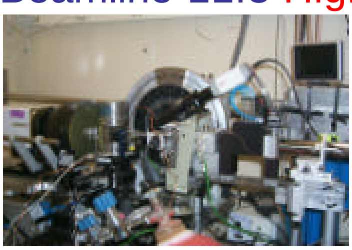
Beamline 12.2.2 is a state of the art beamline for high-pressure diffraction, x-ray imaging and x-ray spectroscopy. Radiation generated by an ALS superbend is delivered to the experimental enclosure by a set of self aligning brightness preserving optics. A monochromatic beam (5-35keV) of radiation is focused (120x100µm focal spot) to the first end station which is equipped with suitable goniometry and a CCD detector.



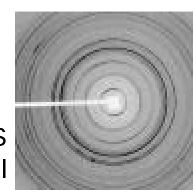
This end station is primarily used for high-pressure diffraction with resistive heating. The x-ray beam diverges from this first focus and is refocused by a set of KB mirrors to the second end station (10x10µm focal spot). Here we have a double sided laser heating system, on-line Raman and ruby fluorescence. The second end station is equipped for x-ray diffraction, x-ray imaging and x-ray spectroscopy.

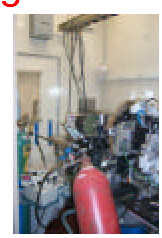
M. Kunz, A.A. MacDowell, W.A. Caldwell, D. Cambie, R.S. Celestre, E.E. Domning, R.M. Duarte, A.E. Gleason, J.M. Glossinger, N. Kelez, D.W. Plate, T. Yu, J.M. Zaug, H.A. Padmore, R. Jeanloz, A.P. Alivisatos, and S.M. Clark. *J. Synch. Rad.* **12(5)** 650-658 (2005).

Beamline 11.3 High-pressure x-ray diffraction with resistive heating

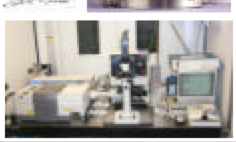


Beamline 11.3 receives focused (120x100µm focal spot) monochromatic (16keV) x-radiation from an ALS bend magnet. It is equipped with suitable goniometry, an on-line viewing system and a Bruker CCD detector. Twenty per cent of the time on this beamline is assigned to COMPRES users. It is primarily used for diffraction from diamond anvil cells with resistive heating.

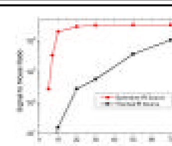


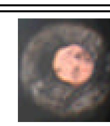


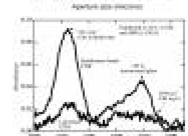
Beamline 1.4 High-pressure infrared spectroscopy



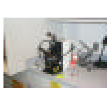
High-pressure infrared data are collected on beamline 1.4. The infrared beam is focussed to a 10µm spot. Spectra are collected using a Nicolet Magna 760 Nic-Pan microscope and FTIR spectrometer. The system has a resolution of about 4 cm^{-1}. Two low profile (21mm thick) diamond anvil cells (one equipped with resistive heating) are available for use on this beamline. Example spectra from a sample of talc are shown on the right.

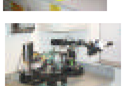




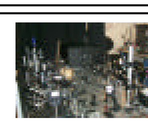


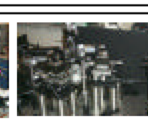
High-Pressure Laboratory

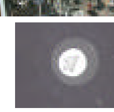




The high pressure laboratory contains all of the equipment necessary for loading and maintaining diamond anvil cells and rudimentary sample preparation. This includes microscopes, micro-drill and a micro-spark eroder. The laboratory also is equipped with Raman and a Brillouin systems suitable for high-pressure measurements.







Photographs of theBrillouin system and a spectrum collected from a cubic Boron Nitride crystal

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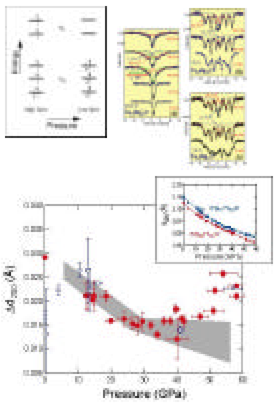
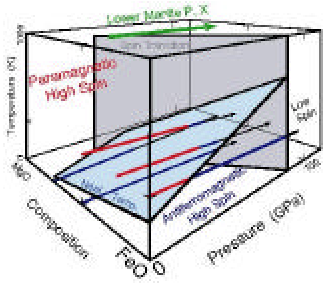
West Coast High-Pressure Facilities Science Highlights

X-ray diffraction: A study of the high-low spin transition in $Mg_{1-x}Fe_xO$

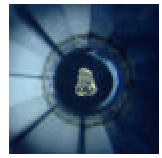


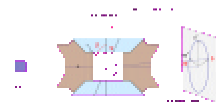


High-pressure Mössbauer spectroscopy on several compositions across the (Mg,Fe)O magnesiowüstite solid solution confirmed that ferrous iron (Fe2+) undergoes a high-spin to low-spin transition at pressures and for compositions relevant to the bulk of the Earth's mantle. High-resolution x-ray diffraction measurements on beamline 12.2.2 found a volume change of 4–5% across the pressure-induced spin transition, which is sufficient to cause seismological anomalies in the lower mantle. The spin transition can lead to dissociation of Fe-bearing phases such as magnesiowüstite, and it reveals an unexpected richness in mineral properties and phase equilibria for the Earth's deep interior.

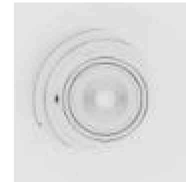
S. Speziale, A.A. Milner, V.E. Lee, S.M. Clark, M.P. Pasternak and R. Jeanloz, **Spin Transition in Earth's Mantle,** *PNAS*, **102(50)** 17918-17922 (2005).

Radial Diffraction: A study of copper to 30GPa

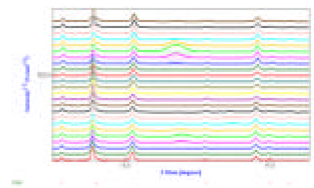


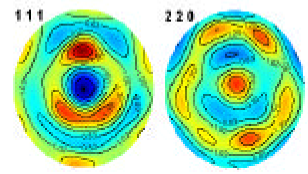


From S. Merkel



The combination of the diamond-anvil cell, synchrotron x-ray diffraction in radial geometry and simultaneous Rietveld refinement with texture analysis allows the quantitative investigation of the plastic deformation behaviour of materials at very high pressures.

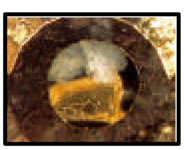


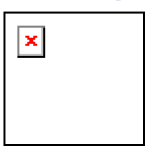


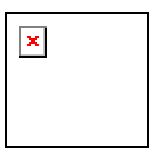
This study of copper to 30 GPa shows in ideal experimental geometry a [110] fibre texture component, as is typical for axial compression of soft face centred cubic metals. Locally a plane strain texture develops which is energetically favoured (curling). A transition from compressional to plane strain/pure shear texture was monitored by analysing individual images taken at different positions in the diamond cell.

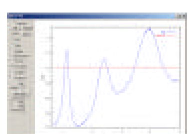
S. Speziale, I. Lonardelli, L. Miyagi, J. Pehl, C. Tommaseo, and H-R. Wenk **Deformation experiments in the diamond-anvil cell: Texture in Copper to 30 GPa**, *J. Phys.: Condens. Matter* **18** S1007-S1020 doi:10.1088/0953-8984/18/25/S08 (2006).

Amorphous and liquid scattering: Phase transition in amorphous red phosphorus

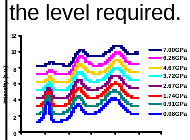


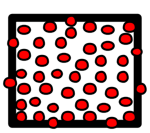


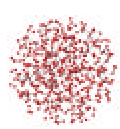




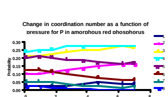
Understanding the effect of pressure on the structure of liquids and amorphous materials is essential for the understanding of the deep Earth. The lack of long range order makes it difficult to determine structural to the level required.

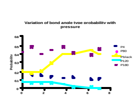


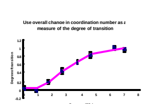




Here we combine x-ray diffraction data collected on beamnline 12.2.2 from a sample of amorphous red phosphorus collected at a range of pressures together with inverse-Monte Carlo modelling.



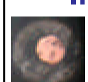


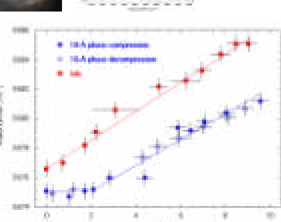


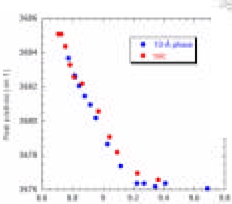
We find that the data are sufficient for us to be able to determine an average structure for the amorphous phosphorus. Changes in the coordination number and bond angles indicate a phase transition at 3GPa.

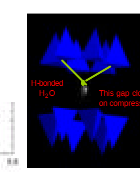
S.M. Clark, J. Zaug and A.L. Soper, **Amorphous-amorphous phase transition in red phosphorus,** *In Prep.* (2006).

Infrared: The role of water in mineral stability









The effect of pressure on the O-H stretching frequencies of natural talc and two samples of 10-Å phase was measured on beamline 1.4 at pressures up to 9.6GPa.The O-H stretching vibration of Mg_3OH groups in talc increases linearly with pressure at 0.97(2) cm^{-1} GPa^{-1}. The same vibration occurs in 10-Å phase, but shows negligible pressure shift up to 2 GPa, above which the frequency increases linearly at 0.96(3) cm^{-1} GPa^{-1} (10Å-46) and 0.87(3) cm^{-1} GPa^{-1} (10Å-160). Other bands in the 10-Å phase spectrum are due to stretching of interlayer H2O, hydrogen-bonded to the nearest tetrahedral sheet. These bands also show little change over the first 2 GPa of compression. Most of the compression of the structure is taken up by closing non-hydrogen bonded gaps between interlayer H2O and tetrahedral sheets. Above 2 GPa, increased hydrogen bonding causes the interlayer vibrations to shift to lower frequency. At the same time, two additional bands are resolved and increase in intensity to 4.4 GPa, suggesting a rearrangement of the hydroxyl groups.

A. Pawley, S. Parry and S.M. Clark, **IR study of the effect of pressure on talc and 10A phase**, *Am. Min.* In Press (2006).

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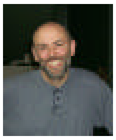
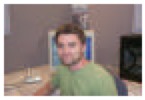
West Coast High-Pressure Facilities
Usage and future developments

West coast people

Raymond Jeanloz
Earth and Planetary Sciences
UCB Berkeley
Principle Investigator

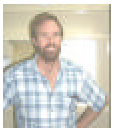


Simon Clark
Advanced Light Source
Calipso project manager
Co-PI

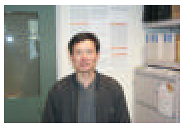


Robin Titus
Advanced Light Source
Associate Beamline Scientist

Martin Kunz
COMPRES
Beamline Scientist

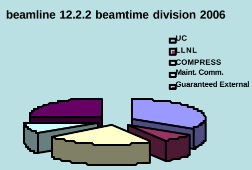


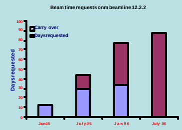
Sander Caldwell
COMPRES
Associate Beamline Scientist

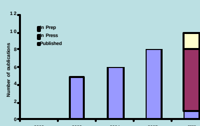


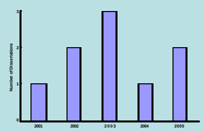
Jinyuan Yan
COMPRES
Post-Doc
Automation of data analysis

Beam time take up







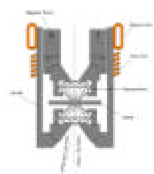


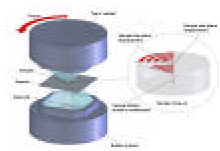
User origins

Red circles show the locations of COMPRES user groups and blue circles the locations of non-COMPRES user groups

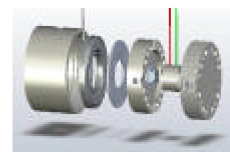
New types of diamond anvil cell

A bellows driven cell for radial diffraction designed to allow laser heating while collecting data



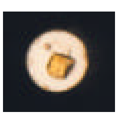


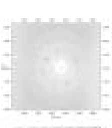
A rotational diamond anvil cell that allows independent application of hydrostatic pressure and shear stress



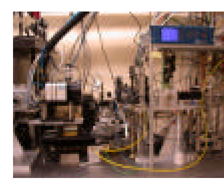
Resistively heated diamond anvil cells combining internal and external resistive heaters.

Single crystal diffraction





X-ray diffraction from single crystal is the best method for structure solution and refinement. At the moment there is no dedicated facility for single crystal measurements at high-pressure in the United States. We are currently converting end station 1 of beamline 12.2.2 for single crystal measurements with the addition of suitable goniometry. This will allow us to measure accurate structural information in a diamond anvil cell (DAC) up to ~ 60- 70 GPa: This will give atomic coordinates of sufficient quality to allow the determination of reliable bond lengths (? d/d ~ 10^{-3}) and angles (? angle/angle ~ 10^{-2}) and isotropic displacement parameters. This will allow us, for example, to determine the exact level and mode of distortion for $MgSiO_3$ perovskite, the most important mineral of the lower mantle. It will also allow us to determine orientation matrices of crystals mounted in a DAC for experiments sensitive to crystal orientation (e.g. Brillouin spectroscopy) and to obtain the most accurate cell parameters for equation of state measurements.



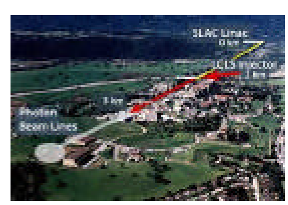
Before

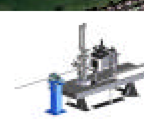
After

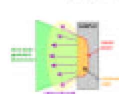
Shockwave measurements

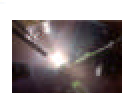
Diamond anvil cells are our main method for the study of materials at high pressure and temperature. Diamond cells are limited to relatively modest temperatures and pressures, have a very small sample volume, have restricted x-ray access, provide poor sample containment at high pressure and temperature and x-ray absorption by diamonds limits the minimum wavelength that we can use and hence limits the available absorption edges for, say, anomalous scattering. Shoc kwaves can be used to generate high pressures and temperatures, and might provide an alternative route to determining crystal structures under extreme conditions. Unfortunately, the shocked state is only stable for a few nano-seconds making it difficult to use x-ray diffraction for structural studies. The Linac Coherent Light Source being built at the Stanford Linear Accelerator Center will allow us to collect a whole diffraction pattern using a single 120fs burst of x-rays. We are planning to develop the necessary facilities at LCLS for structural studies relevant to the COMPRES community as part of the warm condensed matter program.

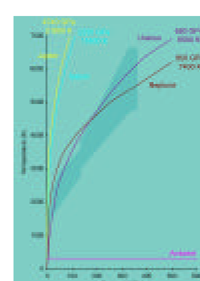
S.M. Clark and R.Jeanloz, **A new paradigm to extend diffraction measurements beyond the megabar regime** *J. Synch. Rad.* **12(5)** 632-636 (2005).











Current techniques

Shockwave techniques